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Composite polymeric membrane with entrapped TiO_2 nano-sized particles for water treatment: optimized elaboration through a structural and functional characterization

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Recent studies have shown that micro-pollutants like pharmaceutics and endocrine disruptors can be found in surface, ground and even potable waters since their elimination by conventional process in water treatment plants is only partial. Their effect on the endocrinal system can be disastrous, even at very low concentration. Several studies have shown that these organic pollutants can be treated using a photocatalytic membrane reactor. Thanks to an UV irradiation, organic pollutants are transformed, using an oxidant catalyst (like titanium oxide nano-sized particles – TiO_2) into non-toxic mineral compounds. The ultrafiltration membrane is used to maintain oxidant in reactor and separate the new product from the catalyst. However, a limitation can be the fouling of the membrane.

In order to limit TiO_2 agglomeration in the reactor but also TiO_2 loss in the permeate, the nanoparticles can be directly entrapped into the polymeric matrix of the membrane during the membrane elaboration process. In addition to be involved in the photocatalytic degradation of the pollutants, TiO_2 could also improve the structural and functional properties of the membrane like mechanical resistance or flow rate performances. For example, an increase of the membrane surface hydrophilicity thanks to TiO_2 could reduce the impact of the organic macro-molecules fouling on the membrane.

The present work aims to develop and study the elaboration process of these polymeric-TiO_2 composite new membranes. The operating parameters and conditions of the elaboration process have a strong impact on the membrane structuration and thus on the membrane performances. In this study, a membrane elaboration process protocol has been successfully developed and its parameters are being optimized in relation with the properties of the elaborated membrane.

Using the phase inversion separation method, the elaboration process protocol has allowed obtaining porous composite membranes. Following this protocol, TiO_2 nano-sized particles (20 nm) are added to the initial polymeric-solvent casting solution and then an ultrasonic treatment of this solution is performed. Solution is then cast on a support and immersed into a deionised water coagulation bath. Intrusion of the aqueous non-solvent into the casting solution induces the phase separation. Different key operating parameters have been studied like the type and presence of additives; the TiO_2 concentration, dispersion or pre-treatment. For each parameter, a similar polymeric membrane without TiO_2 was elaborated. The membranes, with or without entrapped nano-sized particles were characterized in order to determine the influence of the operating parameters on their structural properties (porosity, pore size distribution, thickness, TiO_2 dispersion on the membrane surface and inside the membrane, hydrophilicity) and performances (permeability measurement).

First results and characterization have shown the potential interest of the elaborated porous membranes. Contact angles were measured by the water liquid drop method and were all comprised between 85 and 95°. A key parameter is the dispersion of the TiO_2 inside the casting solution which will have a strong impact on the TiO_2 dispersion in the elaborated membrane and thus on its properties and performances. Best results were obtained using an ultrasonic treatment of the initial casting solution containing the TiO_2. No chemical pre-treatment of the nanoparticles was necessary. Fig 1b and 1c show the repartition of TiO_2 respectively inside the cross-section and on the surface of the composite polymer-TiO_2 membrane. The ultrasonic

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treatment prevents the formation of TiO_2 aggregation and allows the elaboration of the membrane with a good dispersion of the TiO_2 not only on the membrane surface but also inside the membrane pores.

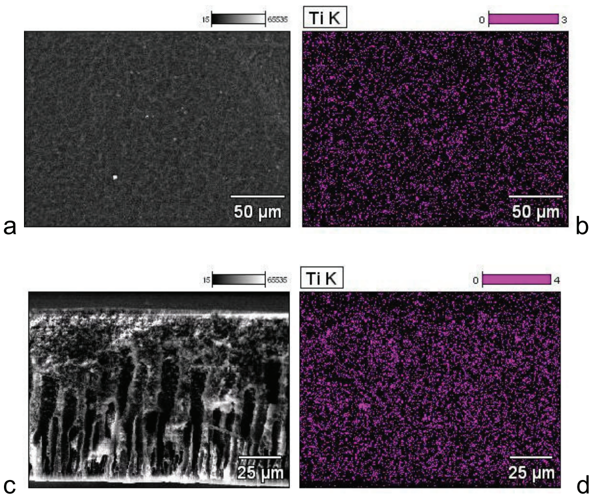

Fig.1 Observation of composites membranes: a)b) Surface SEM (a) and EDS (b) photographs ; c)d) Cross-sectional SEM (c) and EDS photographs (d)

Figure 1a shows the typical structure of the composite membranes. Membranes have a mean thickness of 76 □m. Two different layers are observed with a different structure. The first layer is a thin dense micro-porous layer which allows the separation whereas the second layer is wider and shows finger-like structure which allows maintaining high permeate flux.

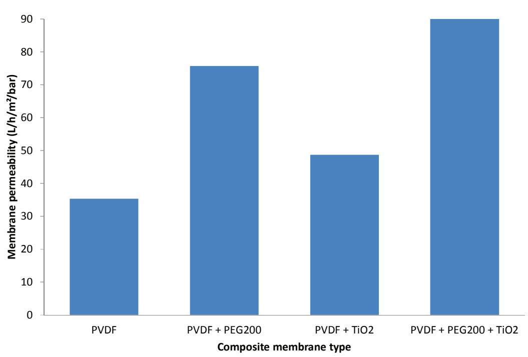

Fig.2 Composite membrane mean permeability for a) PVDF membrane ; b) PVDF membrane with PEG200; c) PVDF membrane with TiO_2 and d) PVDF membrane with PEG200 and TiO_2

Figure 2 shows the permeability of the composite membrane with and without TiO_2 and also with or without addition of PEG200 (Polyethylene glycol which is known to improve membrane permeability). These first permeability tests indicate that the addition of TiO_2 does not reduce the membrane permeability in comparison with polymeric membrane without nanoparticles and might even increase this permeability.

These results must be confirmed by further characterizations and performance tests which are currently underway to precise effects of the elaboration parameters on the membrane properties (pore size distribution, porosity…). As TiO_2 is a photocatalytic agent, influence of an irradiation of the membranes by UV on its performances (photocatalytic activity and permeability) will also be studied.

Keywords: Composite membrane, Nanosized particles, Water treatment